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SIXTH QUARTERLY REPORT

# PRODUCTION ENGINEERING MEASURE FOR SUBMINIATURE TEMPERATURE-COMPENSATING CERAMIC CAPACITORS

PERIOD: 13 SEPTEMBER 1962 THROUGH 12 DECEMBER 1962

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U. S. ARMY SIGNAL SUPPLY AGENCY AST PHILADELPHIA, PENNSYLVANIA

SPRAGUE ELECTRIC COMPANY

NORTH ADAMS, MASSACHUSETTS

# PRODUCTION ENGINEERING MEASURE

#### FOR SUBMINIATURE TEMPERATURE-COMPENSATING

# **CERAMIC CAPACITORS**

# Sixth Quarterly Report

Period: 13 September through 12 December 1962

Object of Study: To establish facilities and competence to produce subminiature temperature-compensating ceramic capacitors

Contract No. DA-36-039-SC-85996 Order No. 6021-PP-61-81-81

# Controlling Specifications:

A .

Signal Corps Technical Requirements SCL-6415, 6 February 1961 Signal Corps Technical Requirements SCS-107, 24 February 1961 Specification MIL-C-20, 11 September 1959

Report Prepared by:

J. H. Fabricius

J. H. D. Folster

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#### **ABSTRACT**

The temperature coefficient of capacitance requirement for the NP0 formulation has not yet been achieved. Approaches attempted during this period failed to produce improved results. Details are provided herein. New studies are now under way.

Meanwhile, pilot-run production on Case Size I and Case Size II units of the N750 and N1400 formulations has begun. Pilot-run production of the NP0 units, however, is being held up pending resolution of the temperature coefficient of capacitance difficulties.

A description of the work done during this period on the development of spraying and dipping slips made with the N1400 formulation and the preferred binder is presented. The achievement of satisfactory dipping slips with the N1400 formulation and the alternate binder is also reported.

Final qualification test results on Case Size II and incomplete test results on Case Size III are presented.

#### **PURPOSE**

# The purpose of this contract is as follows:

- (1) To provide the production engineering to establish capability to manufacture subminiature temperature-compensating ceramic capacitors ranging from 22 μμf-100 VDC to 6800 μμf-100 VDC ratings on a pilot-run basis.
- (2) To design, develop, procure, or manufacture special tooling required for successful pilot-run production.
- (3) To obtain limited production equipment necessary to manufacture 3000 units per eight-hour shift.
- (4) To produce and submit for approval to the Signal Corps preproduction samples prior to the initiation of the pilot run.
- (5) To manufacture a pilot run of 3600 subminiature temperature-compensating ceramic capacitors.
- (6) To provide monthly and quarterly progress reports.
- (7) To prepare production engineering measure final reports in accordance with Step II of SCIPPR No. 15, Paragraph 3.8.

#### NARRATIVE AND DATA

#### 3.1 Selection of Ceramic Materials

Little success has been experienced in the effort to achieve Characteristic CF, the temperature coefficient of capacitance (TC) requirement for the NP0 material. An attempt was made to meet the requirement by calcining the mix before making it into ceramic parts, but this method proved unsuccessful. Firing in a gas kiln was tried but also failed.

Typical data on disc ceramics generated by the calcining study are shown in Table 1. It can be seen that the present capability is far short of that required for Characteristic CF. It does, however, meet Characteristic CG.

Another example of the type of problem being encountered in this study is shown in Table 2, which is a comparison of the TC's of ceramics in disc form with the TC's of the same ceramics in Case Size I Monolythic form. There is a wide divergence in results. It will be noted that in Table 2, the TC's for ceramics in Case Size I Monolythic form, when determined by measuring four units in parallel, are significantly better than those determined by measuring single units. This is because the values of capacitance are extremely small and accurate measurement is difficult with present equipment. However, measurements in single units will be attempted with new equipment, and better results are anticipated.

In Monolythic form, ceramics exhibit a negative temperature coefficient of capacitance, relative to the disc form. It is therefore necessary to produce in disc form a slightly positive TC. To date, this has not been possible. The effort is continuing.

TABLE 1

One Megacycle/Second Capacitance Change with Temperature From Capacitance at 25°C for Precalcined NP0 Formulations And Characteristics CF and CG (Parts per Million)

Designation	Limits	-55°C	-40°C	-10°C	+45°C	+65°C	+85°C
Characteristic	Max.	2800	2000	900	300	600	900
CF	Min.	-900	-800	-500	-300	-600	-900
Characteristic	Max.	4300	3400	1700	600	1100	1700
CG	Min.	-2200	-1900	-1100	-700	-1400	-1800
Body No. 368	Max.	1724	1308	954	-355	-721	-1164
(2360°F Firing)	Min.	1455	990	880	<b>-4</b> 03	-770	-1320
Body No. 368	Max.	1262	1058	1368	-132	-732	-1013
(2390°F Firing)	Min.	1172	989	1118	-256	-769	-1190
Body No. 368	Max.	632	598	858	-381	-538	-837
(2410°F Firing)	Min.	562	596	622	-406	-655	893
Body No. 369	Max.	1864	1230	888	-487	-791	-1338
(2360°F Firing)	Min.	1472	1022	731	-572	-889	-1388
Body No. 369	Max.	1 302	2405	1142	-307	-806	-1253
(2390°F Firing)	Min.	1270	1290	684	-317	-823	-1258
Body No. 369	Max.	1444	1137	1207	-414	-769	-1338
(2410°F Firing)	Min.	1256	1018	865	-639	-794	-1361
Body No. 370	Max.	1810	1219	1271	-382	-690	-1195
(2360°F Firing)	Min.	1671	1211	1084	-387	-776	-1247
Body No. 370	Max.	1162	1104	1930	-231	-738	-1165
(2390°F Firing)	Min.	1141	1065	884	-326	-740	-1283
Body No. 370	Max.	755	839	635	-396	-528	-1067
(2410°F Firing)	Min.	407	742	395	-419	-730	-1233
Body No. 371	Max.	1 329	1179	911	-437	-585	-1081
(2360°F Firing)	Min.	1276	1159	659	-488	-680	-1256
Body No. 371	Max.	••	1108	940	-181	-662	-1145
(2390°F Firing)	Min.	1104	1019	680	-279	-801	-1189
Body No. 371		759	771	533	-510	-688	-1032
(2410°F Firing)		,	• • •		- 3 . V	-000	-1036

One Megacycle/Second Capacitance Change with Temperature
From Capacitance at 25°C for NP0 Material in Disc Form and in Case Size I
Monolythic® Form and Characteristics CF and CG
(Parts per Million)

TABLE 2

Designation	Limits	-55°C	-40°C	-10°C	+45°C	+65°C	+85°C
Characteristic	Max.	2800	2000	900	300	600	900
CF	Min.	-900	-800	-500	-300	-600	-900
Characteristic	Max.	4300	3400	1700	600	1100	1700
CG	Min.	-2200	-1900	-1100	-700	-1400	-1800
Disc Form	Max.	1258	1262	1180	-228	-538	-938
	Min.	745	760	600	-463	-854	-1219
Case Size l	Max.	4823	3663	1893	-319	-1 245	-1795
(Firing No. 1)	Min.	2266	2086	390	-1079	-2105	-2734
		*1811	1453	825	-448	-968	-1659
Case Size 1	Max.	2186	1744		-601	-2051	-2131
(Firing No. 2)	Min.	1641	1070		-923	-2299	-2353
Case Size 1	Max.	2439	2105	1359	-732	-1045	-2892
(Firing No. 3)	Min.	1903	1592	346	-1096	-1696	-3460

<sup>\*</sup>TC's determined from capacitance measurements of four units in parallel.

Other new experiments are now being conducted, also with the aim of meeting Characteristic CF. In particular, there is a study of the effects of using a powder of smaller particle size than used formerly, and of the effects of varying the ceramic compositions made with this powder. There are no results to report on this study yet.

# 3.2 Development of Spraying Capability

The current spraying capability is as follows:

- (1) Spraying slips of the NPO and N750 formulations made with the preferred binder system are satisfactory.
- (2) Spraying slips of the N1400 formulation made with the alternate binder system are satisfactory.

Work is continuing toward developing a satisfactory spray made with the Nl 400 formulation and the preferred binder. As reported in the Fifth Quarterly Report, work toward this end centers around two approaches. One method involves processing the powder in a special manner before making it into a spraying slip. The other method employs oxalate adsorption and/or reaction to promote a negative surface charge on one of the inert ceramic ingredients in the formulation.

In addition, study of a third approach will begin shortly. Details of this investigation will be given after the actual experimentation is under way.

# 3.3 Development of Dip Method

As reported previously, satisfactory dipping slips made with the preferred binder system have been achieved for the NPO and N750 formulations. Capability has also been attained for producing satisfactory slips made with the alternate binder for the NI400 formulation.

Effort is continuing toward developing a dipping slip made with the N1400 formulation and the preferred binder.

#### 3.4 Qualification Test Samples

# 3.4.1 Case Size II

Qualification test results from Test Groups I-IV of the Case Size II units (Contract Item 1-3-6) were reported in the Fifth Quarterly Report. Results from the final group are given below:

# Test Group V

All testing is completed. One unit exhibited a capacitance change which was out of tolerance following life test. All other units passed. Test reports and samples were submitted to the U. S. Army Signal Supply Agency on September 20, 1962.

# 3.4.2 Case Size III

Test results on the 128 Case Size III units undergoing qualification testing (Contract Item 1-3-9) are summarized below:

# Test Group I

All testing is completed. One unit exhibited low insulation resistance. All other units passed.

#### Test Group II

All testing is completed. One unit exhibited low insulation resistance following the temperature immersion test. All other units passed.

#### Test Group III

All testing is completed. All units passed.

#### Test Group IV

All testing is completed. All units passed.

#### Test Group V

Testing will be completed February, 1963.

# 3.5 Pilot-Run Production

Pilot-run production on Case Size I of Formulations N750 and N1400 (Contract Items 1-3-2 and 1-3-3, respectively) and on Case Size II of Formulations N750 and N1400 (Contract Items 1-3-5 and 1-3-6, respectively) is now under way. Pilot-run production on Case Size I and Case Size II of the NP0 formulation (Contract Items 1-3-1 and 1-3-4, respectively) is being held up pending further efforts toward attaining the temperature coefficient of capacitance requirement.

# 3.6 Technical Action Request

Technical Action Request No. 4, a request to change the Characteristic CF requirement to a Characteristic CG requirement, was disapproved during this period.

# 3.7 Extension of Delivery Date

An extension of the delivery date for the Case Size III qualification samples (Contract Item 1-3-9) was granted during this period (letter of Mary V. Tumelty to Arthur G. Ceely, November 8, 1962). The delivery date for these samples is now February 7, 1963.

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#### CONCLUSIONS

- (1) Efforts toward developing an NPO material capable of complying with TC Characteristic CF in the required case sizes are continuing.
- (2) Satisfactory sprays made with the N1400 formulation and the alternate binder system are now being produced. Meanwhile, work is continuing toward achieving spraying capability with a slip made of the N1400 formulation and the preferred binder system.
- (3) Satisfactory dipping slips made with the alternate binder have now been achieved for the N1400 formulation. Work toward producing a satisfactory slip made with the N1400 formulation and the preferred binder system is continuing.
- (4) Test results from qualification testing of Group V of the Case Size II units of the N1400 formulation are satisfactory.
- (5) Test results from qualification testing of Case Size III units of the N1400 formulation are satisfactory to date.
- (6) Pilot-run production on Case Size I of Formulations N750 and N1400 (Contract Items 1-3-2 and 1-3-3, respectively) and on Case Size II of Formulations N750 and N1400 (Contract Items 1-3-5 and 1-3-6, respectively) is now under way.
- (7) Pilot-run production on Case Sizes I and II of the NPO formulation (Contract Items 1-3-1 and 1-3-4, respectively) is being held up pending further efforts toward attaining the temperature coefficient of capacitance requirement.

(8) The estimated percentages of the overall progress on the major elements of the program are as follows:

,	Factor	Relative Weight	% Completion	Percentage
1.	Production Design Engineering	25	95.0	23.8
2.	Engineering and Design of Special Tooling and Refining Equipment	<u>15</u>	95.0	14.2
3.	Pre-production Sample Approval	<u>15</u>	90.0	13.5
4.	Pilot Run	25	15.0	3.8
5.	Monthly & Quarterly Reports	10	68.8	6.9
6.	Final Report, Step II Study and Inspection	10		
	Test Report	10	0.0	0.0
	TOTALS	100		62.2

# PROGRAM FOR NEXT INTERVAL

- (1) Continued effort will be made toward achieving Characteristic CF for the NP0 formulation. Further study will be made of the effects of a new powder.
- (2) The effort toward producing satisfactory spraying and dipping slips of the N1400 formulation and the preferred binder will continue.
- (3) Qualification testing of the Case Size III units will continue.

#### **PUBLICATIONS AND REPORTS**

The following reports were submitted to the U. S. Army Signal Supply Agency during the quarter:

- (1) The fifth quarterly report, covering the period 13 June 1962 through 12 September 1962, was submitted for U. S. Army Signal Supply Agency approval. Approval was received, and the report was distributed per Signal Corps instructions.
- (2) Monthly reports for September, 1962, comprising narrative reports, progress reports on SA-SC Forms 109-1 and 109-2, and progress percentage calculation.
- (3) Monthly reports for October, 1962, comprising the same.
- (4) Monthly reports for November, 1962, comprising the same.

SECTION 7

# IDENTIFICATION OF PERSONNEL

Personnel	Hours
W. Baker	17.0
J. Dziok	6.3
W. Estes	49.5
J. Fabricius	21.0
R. Fisher	18.5
W. Hatch	23.0
R. Immediato	55.0
T. Jammallo	99.0
E. Jones	7.5
L. Lamore	33.0
J. Ledoux	7.0
L. Lemoine	10.0
C. Moore	40.5
P. Moriarty	79.0
J. Newman	21.0
G. Olsen	3.0
T. Prokopowicz	9.0
D. Reid	20.0
R. Trottier	12.0
K. Whitney	6.0
J. Willey	19.5
M. Zedalis	2.3
Total	559.1

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